

MEASUREMENT OF THE PERFORMANCE OF SOME NEW VAPOUR PHASE ACTIVE FILTERS BY AN IMPROVED GAS CHROMATOGRAPHIC METHOD.

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SUMMARY

The increasing world-wide interest in the use of cigarette filters containing granular adsorbents has led to the need for a rapid method to measure the activities of such materials in cigarette filters. Previously, the vapour phase activity of filters was commonly measured by Gas Chromatography using long packed columns with the associated problems of high bleed rates and the time needed to obtain satisfactory resolutions of the majority of vapour phase compounds. An improved GC method has been developed for the rapid determination of vapour phase constituents in cigarette smoke using the high resolution now available from fused silica bonded phase capillary columns. Relatively undemanding experimental conditions are required, a temperature programme from 40 to 225°C allowing a good separation to be achieved in twenty five minutes. This method has now been in routine use at Filtrona for over two years without any deterioration in column performance.

Two new types of filters have been studied, both of which allow high loadings of granular materials to be achieved. Typical performance data of these filters is reported, showing their effect on the vapour phase of cigarette smoke. A comparison of the use of carbon and sepiolite as adsorbents is also given.

INTRODUCTION

In recent years, there has been an increase in demand for filters containing adsorbents, particularly carbon. Such filters are now in use in many countries, e.g. Italy, Korea, China and of course in Japan. The

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opening of the Japanese market to foreign imports, especially from the U.S.A., has led to major increases in the production of carbon-containing filters. In parallel with the necessary improvements in production techniques to meet this demand, Filtrona have also been developing new types of adsorbent filter.

Testing methods for Vapour Phase active filters include specific compound tests e.g. for formaldehyde and HCN, carried out on whole smoke because they do not conform to the phase partitioning obtained with Cambridge filters. However, for general tests of activity, gas chromatographic separation of organic vapour phase compounds has usually been used. In common with many other laboratories, we used packed column methods for many years, and reasonable resolution could be obtained with carefully selected stationary phases, used at high loadings on columns up to 4 metres long. These methods had the disadvantages of fairly short column life, the necessity of matched column pairs because of high bleed rates, and long analysis times. In recent years, the development of bonded phases on fused silica capillary columns has provided better separating power combined with much greater column stability and lower analysis times.

METHOD

Cigarettes were smoked to tip + 8mm butt through a Cambridge filter holder on a Filtrona SM 302 smoking machine. Eight cigarettes were smoked per analysis the combined vapour phase being collected in a single bag made from biaxially orientated polypropylene/polyester laminate. Samples were analysed on a temperature programmed Carlo Erba Vega GC equipped with a Shimadzu MGS4 gas sampling valve with a 0.5cm^3 sample loop, a flame ionisation detector and a $20\text{m} \times 0.32\text{mm}$ Poraplot Q fused silica capillary column, with a film thickness of $10\mu\text{m}$.

The temperature programme was as follows: (i) 40°C (hold min) to 90°C at a rate of $45^\circ/\text{min}$; (ii) 90°C to 205°C (hold 1 min) at a rate of $5^\circ/\text{min}$; (iii) 205°C to 225°C (hold 5 min) at a rate of $45^\circ/\text{min}$. The detector temperature was 240°C .

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Helium was used as the carrier gas at a flow rate of $2.1\text{cm}^3/\text{min}$ through the column, with a split ratio of 16:1. The GC analysis time was approximately 32 minutes per sample.

Data was collected using a Spectra Physics SP 4290 computing integrator. A typical chromatogram is shown in Figure (1).

All results were the mean of three analyses.

RESULTS

The method has been used in the testing of two new types of carbon filter, both of which have high adsorbent loading capability.

The AMW Filter

Carbon, or other adsorbent granules, are distributed throughout an embossed paper segment. The adsorbent is held to the paper by use of a small amount of thermoplastic polymer, precoated onto the granules, and subsequently heat-treated. High loadings of adsorbent can be achieved, depending on the density: for example, up to about 9mg/mm of activated carbon. Tests were done on $21 \times 24.4\text{mm}$ dual filters with 8mm NWA mouth end segments and 13mm AMW segments, see Figure (2). The filters were assembled to tobacco rods, and tested after several weeks storage. Indirect retentions were calculated by comparison of mean values (3×8 cigs) with those from the tobacco rods alone.

Figure (3) shows retentions for a number of organic VP compounds at a filter loading of 5.02mg/mm , ie a total carbon loading of 65.3mg/filter . As would be expected with carbon, the retentions are fairly similar for different compounds, with a slight tendency to increase with higher boiling compounds such as butanone and toluene.

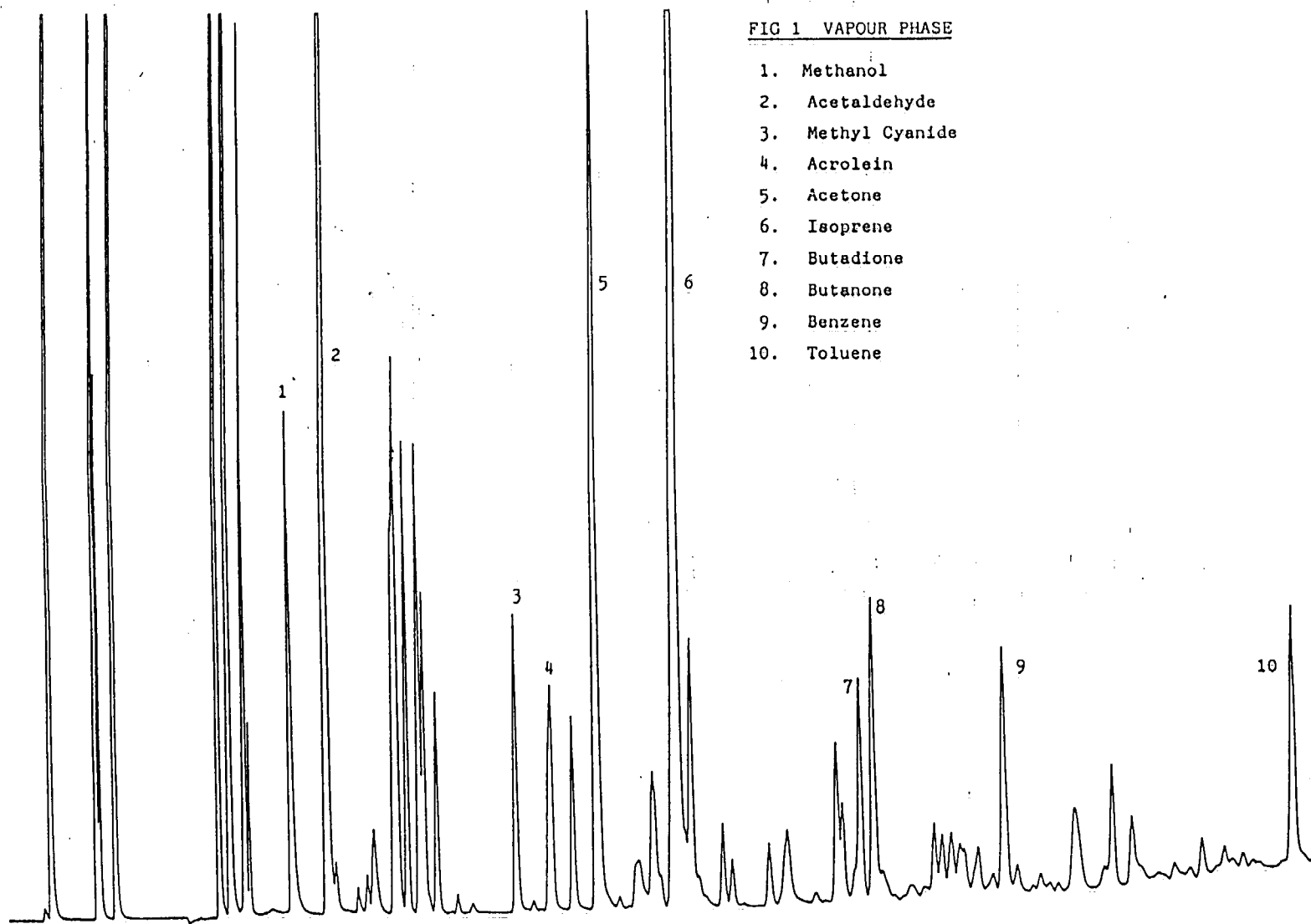
Figure (4) shows the effect of carbon loading for a few selected compounds, and the increases shown are typical of all of the compounds measured.

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The Adsorbent Coated Thread (ACT) Filter

The ACT filter is an entirely new method of presenting granular additives to smoke in a cigarette filter. Depending upon additive loading, a number of threads, coated with carbon and/or other granules are formed into a wrapped filter rod or placed in the axis of an acetate filter. This becomes the inner segment of a dual filter or the centre segment of a triple solid filter as shown in Figure (5). In the triple solid form the carbon is fully encapsulated so that no part containing the additive is cut through during cigarette assembly eliminating loose granules in product or equipment. In use the smoke will pass through the lower pressure drop granular core, thus promoting effective exposure to the active material. Very high loading of active materials can be achieved, up to 15mg/mm depending upon the type and density of the adsorbent used. The ACT filters were tested in the same way as the AMW filters, however, in this case triple solid filters were tested using two 6mm NWA end segments with a 15mm segment of ACT filter in the centre. Figure (6) shows retentions for a number of organic vapour phase components at filter loadings of 5.2mg/mm (sepiolite) and 6.1mg/mm (carbon). That, is total loadings of 78mg sepiolite and 91.5mg carbon. The difference between the two adsorbents is fairly obvious with the polar sepiolite showing selective action towards polar compounds. The carbon loaded filter gives very similar behaviour to that seen previously for the AMW filter. Figure (7) shows the increase in component retention for a few selected compounds with increasing adsorbent levels. Again the increases shown are fairly typical of all the compounds measured at least for carbon. With the highest carbon loaded ACT filter approximately 75% of all vapour phase compounds are removed even using an unventilated cigarette.

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Fig. 2 AMW DUAL FILTER

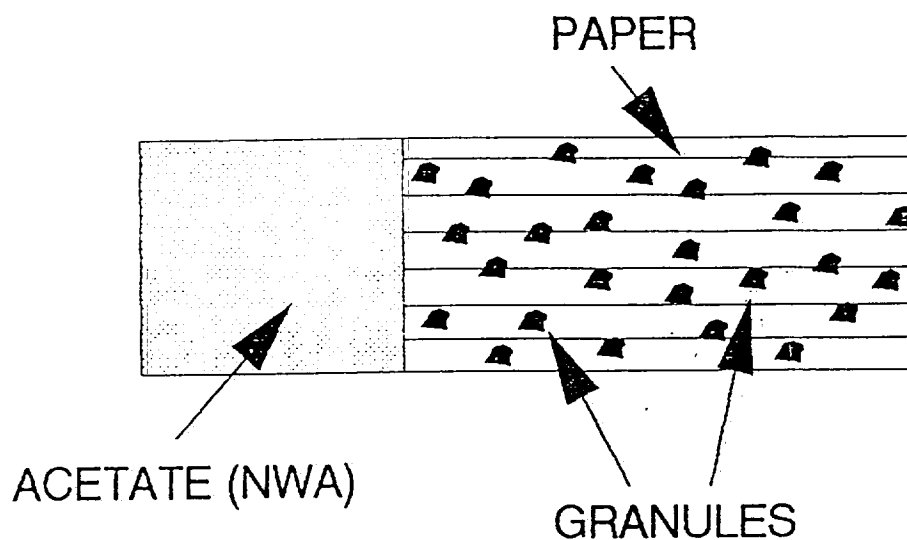
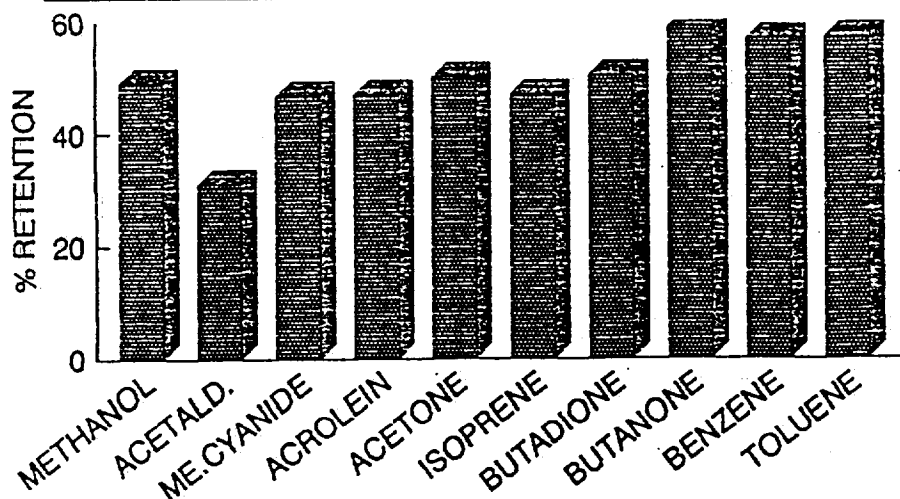


Fig. 3 AMW DUAL : V.P. RETENTIONS



(8 + 13) X 24.4 mm : CARBON 65.3 mg/FILTER

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Fig. 4 AMW DUAL FILTER : EFFECT OF CARBON LOADING

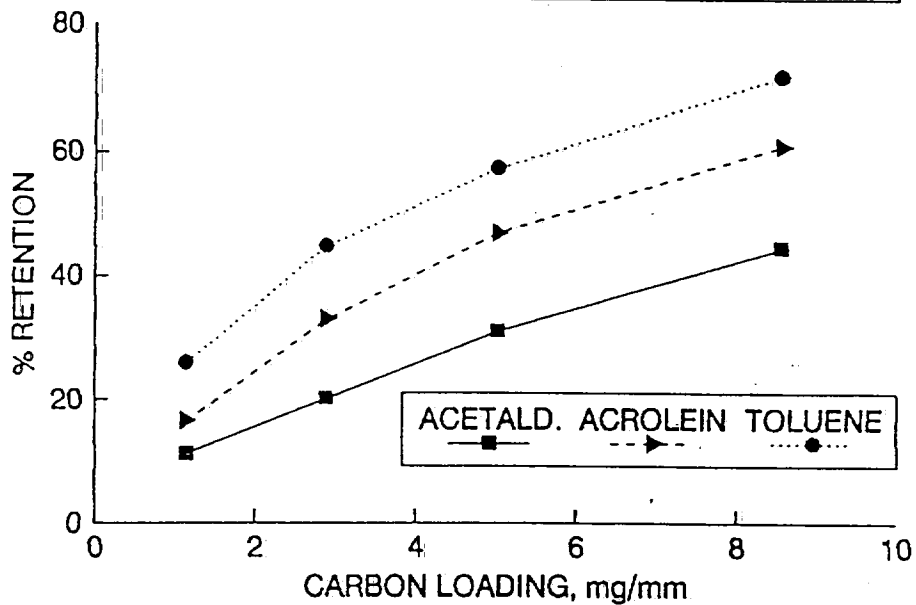
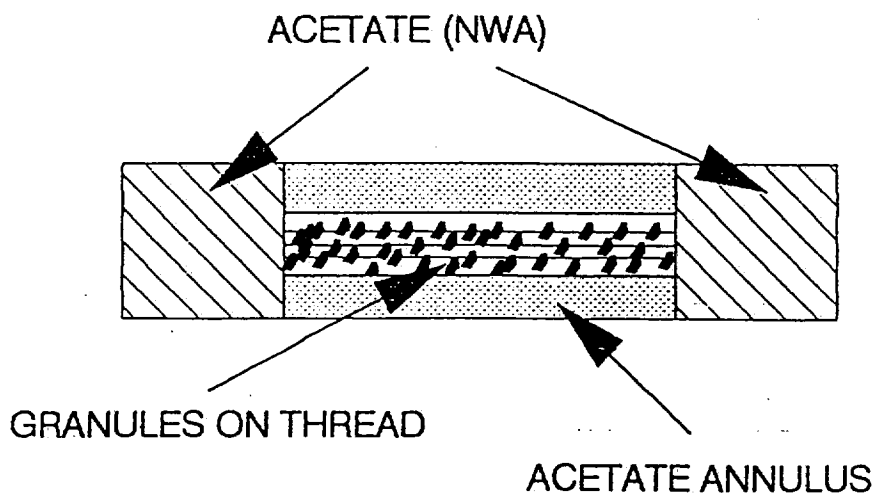


Fig. 5 ACT TRIPLE SOLID FILTER



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Fig.6 ACT TRIPLE : DIFFERENT ADSORBENTS

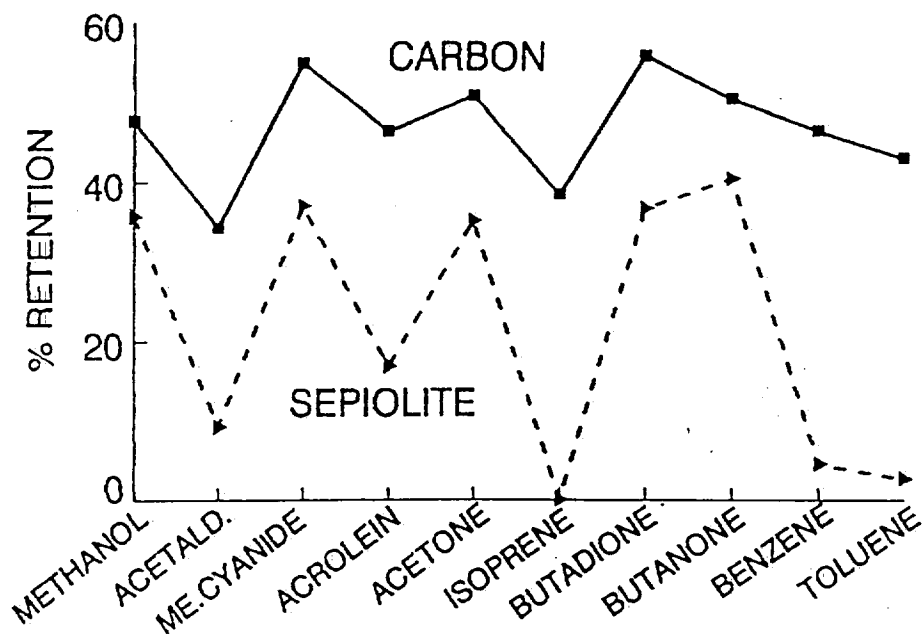
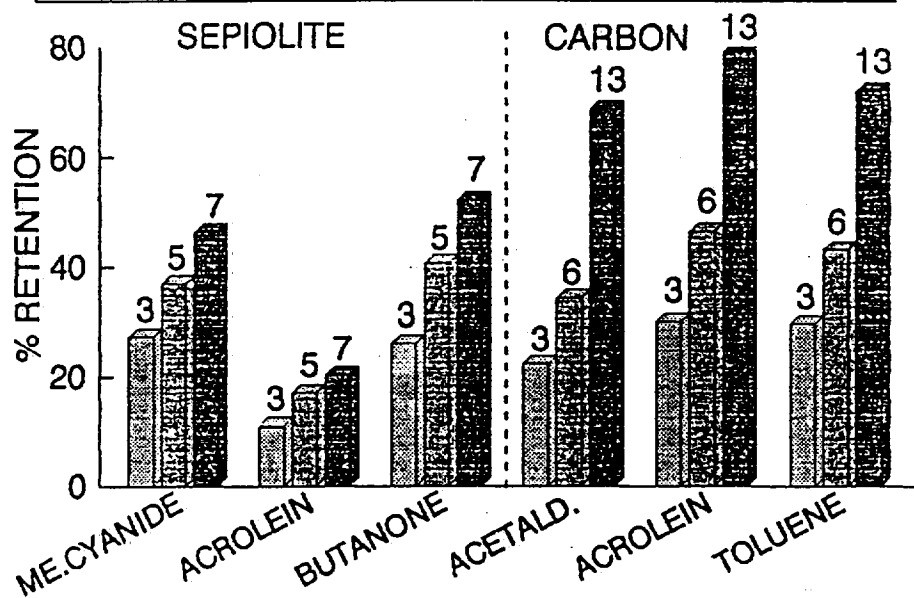


Fig. 7 ACT TRIPLE : LOADING EFFECTS



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